



Ceramic Strain Gages for Use at Temperatures up to 1500 °C

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As the temperature capability of engine components is increased and new materials are developed to meet these new challenges, there is a growing need to assess material behavior in these environments so that structural models can be validated and engine materials can be further characterized. Towards this end, a robust thin film strain gage was developed to monitor both static and dynamic strain of components employed in propulsion systems operating at temperatures in excess of 1480 °C. Specifically, a ceramic strain gage based on alloys of indium-tin oxide (ITO) was developed to meet future materials requirements within NASA's Aerospace Propulsion and Power (APP) programs. These ceramic sensors are capable of in-situ strain measurement in harsh environments such as those encountered in the hot sections of turbine engines without compromising engine performance or operation. The ceramic sensors are refractory, non-intrusive and robust enough to withstand the high "g" loading associated with rotating components. And since the gage thickness is considerably less than the gas phase boundary layer thickness, the gas flow path through the engine will not be adversely affected by these sensors.

Damage assessment of structural components employing advanced ceramic matrix composites (CMC's) is particularly difficult, since conventional strain gage protocols do not lend themselves to these materials. For example, SiC-SiC CMC's are difficult to instrument for damage assessment due to their very rough surfaces and lack of suitable welding techniques for attachment of lead wires to the instrumentation. Thin film ceramic strain gages can be deposited directly onto the surface of these materials and thus offers an alternative solution to structural engineers who must design CMC components for these harsh environments. Alloys based on ITO ceramics exhibit superior chemical stability relative to other strain gage materials in the temperature range 1050–1480 °C and can be deposited directly onto a variety of surfaces by rf sputtering. Due to oxidation and other effects, state-of-the-art PdCr strain gages are limited to temperatures of 1050 °C.

A detailed summary of the accomplishments completed to date under the APP program is presented below and the major tasks (milestones/benchmarks) accomplished within the proposed time frame are highlighted in *italics*. Most significant was the large number of ITO strain gages that were tested under static strain conditions at temperatures up to 1500 °C. The static strain tests were completed under laboratory conditions at the University of Rhode Island and were made using a cantilever bend fixture fabricated out of a zirconium phosphate ceramic (Figure 1). An alumina rod, connected to the constant strain beam, is connected to a linear variable differential transducer (LVDT) to measure deflection of the beam. Corresponding resistance changes were monitored with a 6 1/2 digit Hewlett Packard multimeter in conjunction with a programmable Keithley 7001 switch and a Keithley constant current source. For continuous data acquisition, the instruments were interfaced to an I/O board and an IBM PC employing an IEEE 488 interface and National Instruments LabWindows.

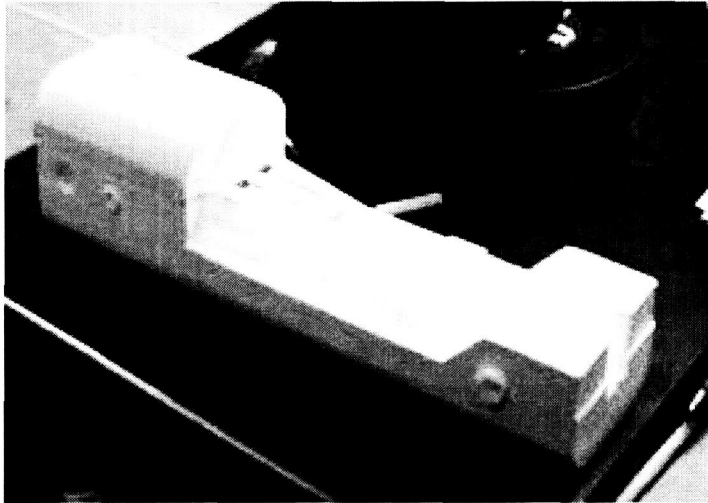


Figure 1. Photograph of the high temperature, zirconium phosphate cantilever bend fixture. Note the alumina constant strain beam clamped in the fixture and the leadwires attached to the sensor.

Static Strain Tests of Sensors on Alumina at Temperatures Approaching 1500 °C

We have demonstrated that ITO thin film strain gages could operate at temperatures approaching 1500 °C. A series of static strain tests of ITO sensors prepared on alumina substrates was performed over the temperature range 25 to 1500 °C. These laboratory tests in air indicated that relatively thick ITO strain gages produced a stable piezoresistive response with little drift over the entire temperature range. These high temperature static strain tests revealed that the piezoresistive response and electrical stability depend largely on the thickness of the ITO films comprising the active strain elements of these sensors. An upper use temperature was also established for a given ITO film thicknesses comprising the active strain element in these sensors. Gages employing thick ITO films (8 μm) were the most stable and responsive of all the gages tested to date; e.g. uncompensated ceramic gages prepared with 8 μm of ITO exhibited a gage factor of 39 and drift rate of 0.0290 at 1455 °C whereas self-compensated ITO strain gages exhibited a gage factor of 2.4 and a drift rate of 0.0092 at 1471 °C.

A representative group of ceramic strain gages having different ITO thicknesses comprising the active strain elements were tested under static strain conditions on high purity aluminum oxide substrates. Some of the test results done under static strain conditions are shown in Figures 2 through 7. A self-compensated ceramic strain gage prepared with an 8 μm thick active ITO element had a relatively large piezoresistive response and extremely low drift rate at 1443 °C is shown in Figure 2. These results were very different from those obtained for very thin ITO films (2.5 μm thick) employed as the active strain elements in a self-compensated ceramic strain gage. When these relatively thin ITO films were used, the piezoresistive response was reasonably large at lower temperatures (1157 °C) but became unstable at temperatures above 1225 °C, as shown in Figures 3a and 3b, respectively. In contrast to this, ITO sensors prepared with

5 μm thick active strain elements were very responsive at temperatures as high as 1438 $^{\circ}\text{C}$ is shown in Figure 4. The latter sensors exhibited a gage factor of 6.8 and drift rate of 0.08 and did not become unstable until temperatures approached 1450 $^{\circ}\text{C}$. However, sensors prepared with 8 μm thick active strain elements did not fail until temperatures reached 1482 $^{\circ}\text{C}$. These self-compensated ceramic sensors were extremely stable and responsive, exhibiting a gage factor of 2.4 and a drift rate of only 0.0092 at 1471 $^{\circ}\text{C}$, as shown in Figure 5.

Based on these results, we have concluded that the thickness of the ITO active strain element plays a significant role in the high temperature stability of these ceramic strain gages. Specifically, these results indicated that both gauge factor and drift rate were affected by the thickness of ITO films comprising the active strain elements. Thicker ITO films were effective oxygen diffusion barriers, slowing the migration of oxygen to the platinum: ITO interface and thus, stabilizing the ohmic contact formed between the platinum and ITO. The diffusion of oxygen through the non-stoichiometric indium-tin-oxide can lead to compensation of the doubly charged oxygen vacancies in the ITO that are responsible for conduction in this wide bandgap semiconductor. Thus, the bulk conductivity of the ITO films can also be affected by oxygen diffusion so that a greater portion of thinner films will be compensated in the presence of oxygen. Consequently, thicker ITO films have a greater current carrying cross sectional area, making it more difficult for diffusing oxygen to reduce the bulk conductivity of the active sensor elements. All things being equal, the uncompensated ITO strain sensors proved to be much more responsive than self-compensated strain sensors when tested at elevated temperatures. Extremely large piezoresistive responses have been observed for uncompensated ITO strain gage as shown in Figures 6 and 7. Here, gage factors of 41 and 39 were observed at temperatures of 1367 $^{\circ}\text{C}$ and 1455 $^{\circ}\text{C}$, respectively.

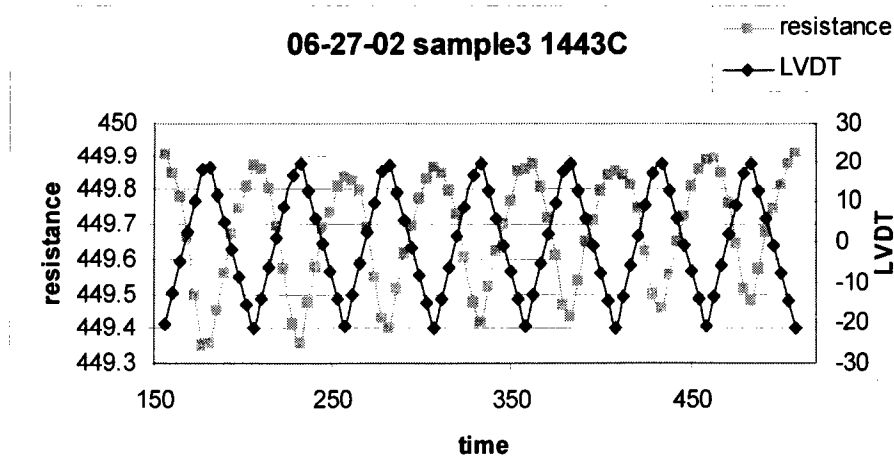


Figure 2. Piezoresistive response of a ceramic thin film strain gage at 1443 $^{\circ}\text{C}$. The thin film gage was deposited on high purity alumina using a bilevel positive resist (polyimide resist) in conjunction with liftoff. The active strain element consisted of an 8 μm thick ITO film and platinum self-compensation circuitry consisting of serpentine arrays with 100 μm lines and spaces. The ITO strain gage exhibited a gauge factor of 20.9 and a drift rate of 0.0001. The sensor ultimately failed at 1482 $^{\circ}\text{C}$.

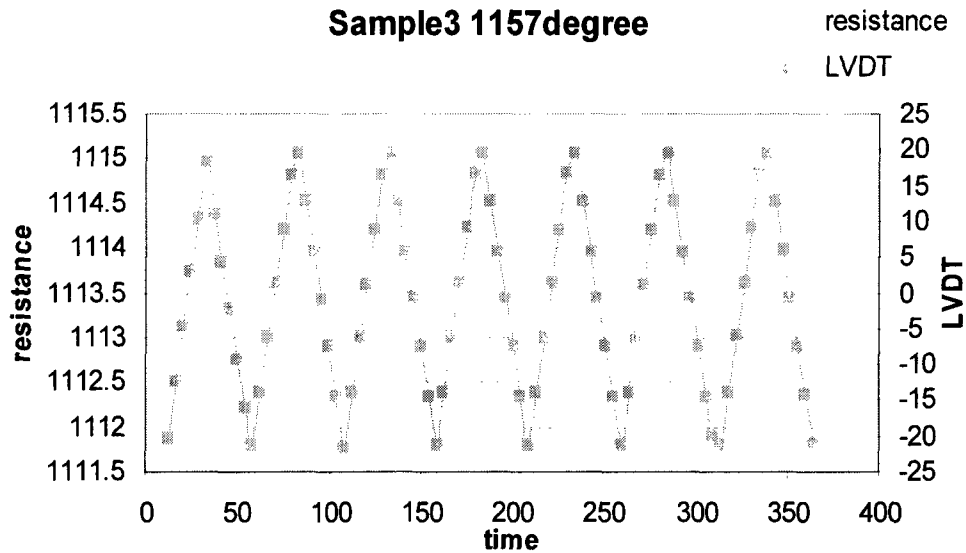


Figure 3a. Piezoresistive response of a ceramic thin film strain gage at 1157 °C. The active strain element consisted of a 2.5 μm thick ITO film and platinum self temperature compensation circuitry (100 μm lines and spaces). Note the piezoresistive response at 1157 °C was relatively large and reproducible. $G = 3.52$, $DR = 0.016$

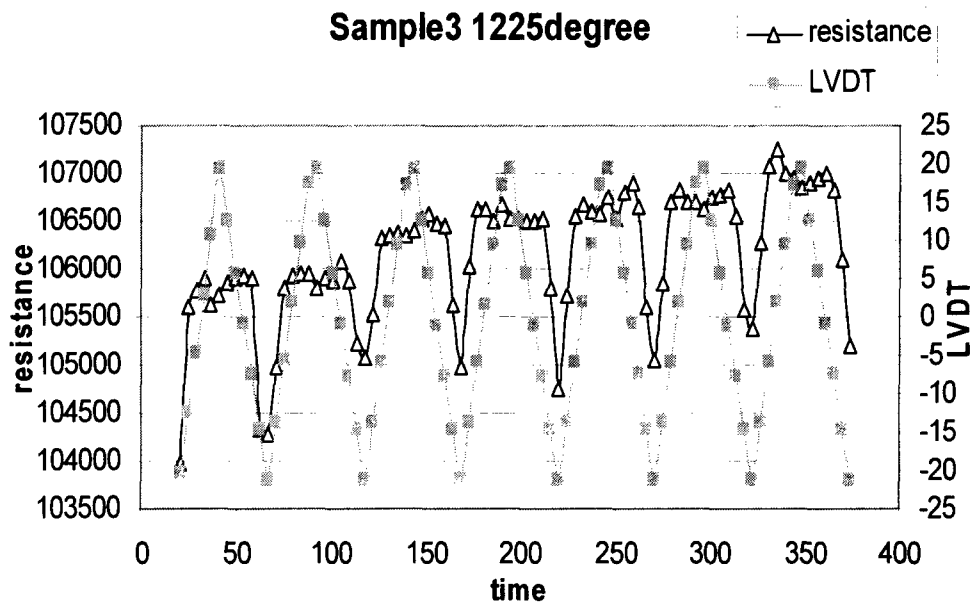


Figure 3b. Piezoresistive response of a ceramic thin film strain gage at 1225 °C. The active strain element consisted of a 2.5 μm thick film of ITO and platinum self temperature compensation circuitry consisting of serpentine arrays with 100 μm lines and spaces. Note the piezoresistive response at 1225 °C begins to breakdown, i.e. the waveform begins to degrade.

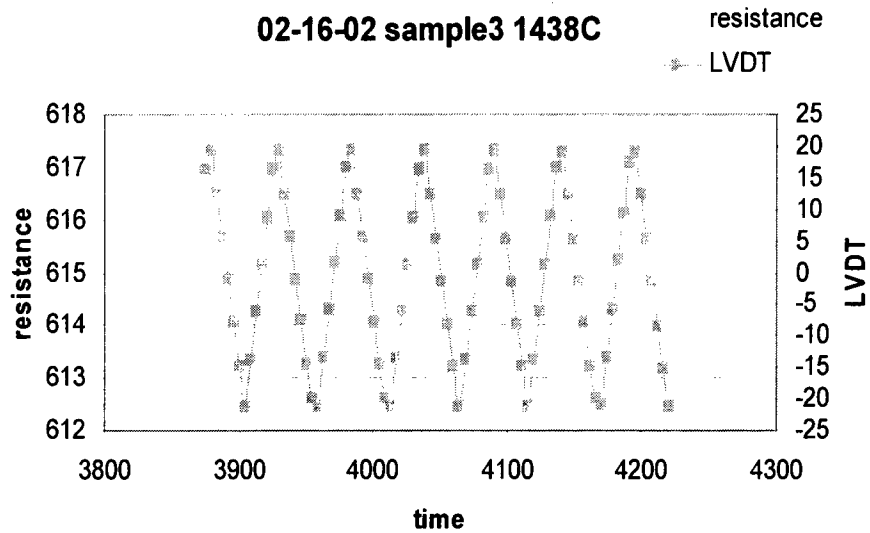


Figure 4. Piezoresistive response of a ceramic thin film strain gage at 1438 °C. The active strain element was based on a 5 μm thick ITO film and platinum self temperature compensation circuitry consisting of serpentine arrays with 100 μm lines and spaces. The ITO sensor exhibited a reasonably large piezoresistive response at temperature, including a gauge factor of 6.8 and drift rate of 0.08.

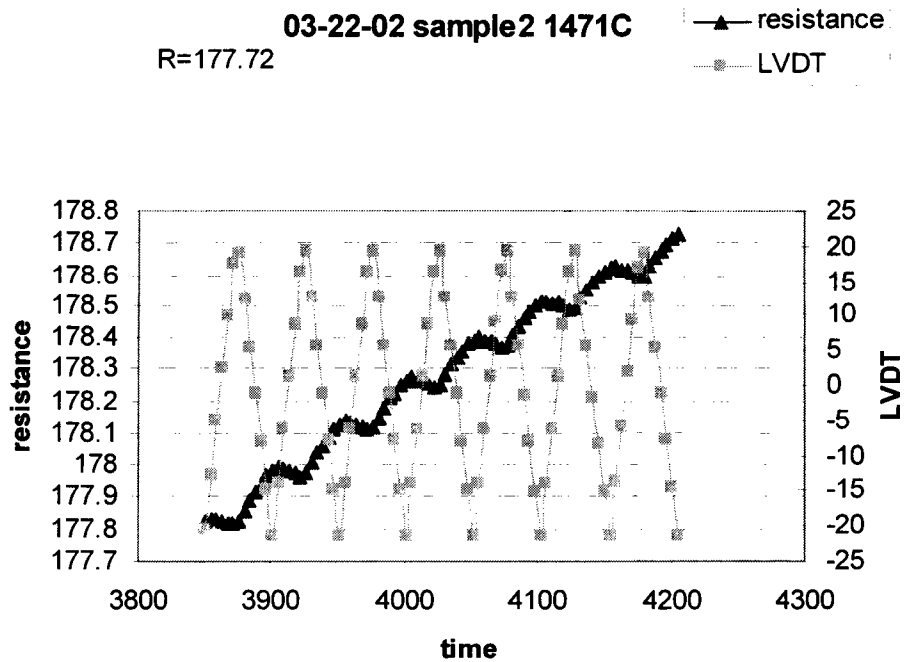


Figure 5. Piezoresistive response of a ceramic thin film strain gage at 1471 °C. The active strain element consisted of 8 μm of ITO and had platinum self temperature compensation circuitry consisting of serpentine arrays with 100 μm lines and spaces. $G = 2.36$, $DR = 0.0092$

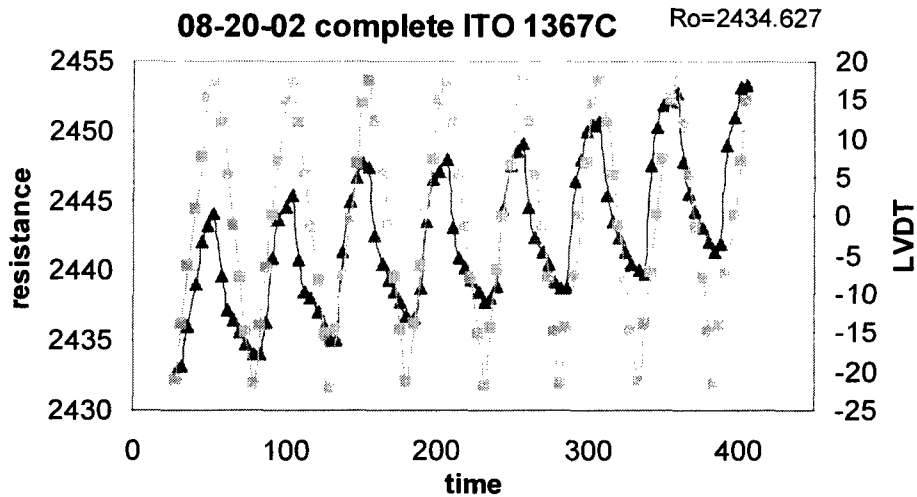


Figure 6. Piezoresistive response of a ceramic thin film strain gage tested at 1367 °C. Note the exceptionally large piezoresistive response and small drift rate at temperature. The active strain element consisted of an 8 μm thick ITO film and did not have platinum self-temperature compensation circuitry. The sensor pattern consisted of serpentine arrays with 100 μm lines and spaces. $G = 41.57$, $DR = 0.035$

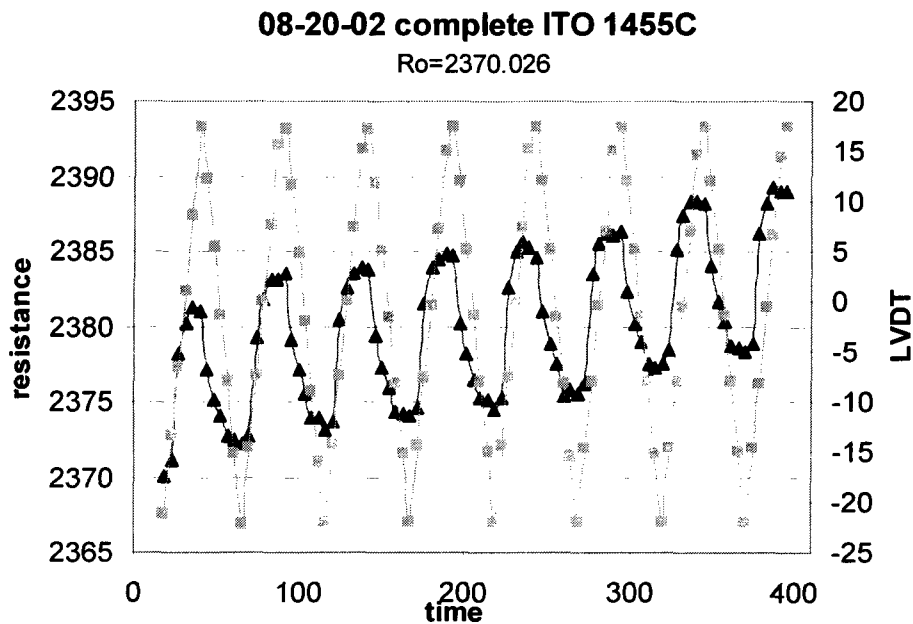


Figure 7. Piezoresistive response of a ceramic thin film strain gage tested at 1455 °C. Note the exceptionally large piezoresistive response and small drift rate at temperature. The active strain element in this sensor consisted of an 8 μm thick ITO film, and had no platinum self temperature compensation circuitry. The sensor pattern consisted of serpentine arrays with 100 μm lines and spaces. $G = 39.07$, $DR = 0.029$

Improved High Temperature Stability of ITO Strain Gages

To further improve the high temperature stability of the ceramic strain gages, the active ITO strain elements were doped with aluminum and heat treated to allow interdiffusion of the aluminum into the surface layers of the ITO. In a series of ESCA (electron spectroscopy for chemical analysis) experiments, we have shown that aluminum plays a significant role in improving the high temperature stability of ITO thin films which provided the motivation for doping the ITO films with aluminum. Recently, we demonstrated that Al doped ITO films were stable in air to temperatures in excess of 1495 °C. Based on ESCA studies, we believe that interfacial reactions between the sputtered ITO films and aluminum containing films were responsible for the enhanced stability of the films at temperatures in excess of those predicted by thermodynamics. To confirm the nature of these interfacial reactions, ESCA depth profiles were performed on ITO films deposited directly onto platinum films and were compared to ESCA profiles of ITO films formed directly on alumina films. The ESCA depth profile in Figure 8 showed that there is indeed an interfacial reaction between the ITO films and alumina, as deduced from the appearance of two new indium peaks (In 3d₅ and In 3d₃ peaks at 449 and 457 eV) as the ITO-Al₂O₃ interface was approached. The two new indium peaks were ~4 eV higher in energy than the corresponding indium peaks in the bulk ITO film. Thus, the higher binding energies (and bond strengths) associated with the indium-indium bonds suggests an improvement

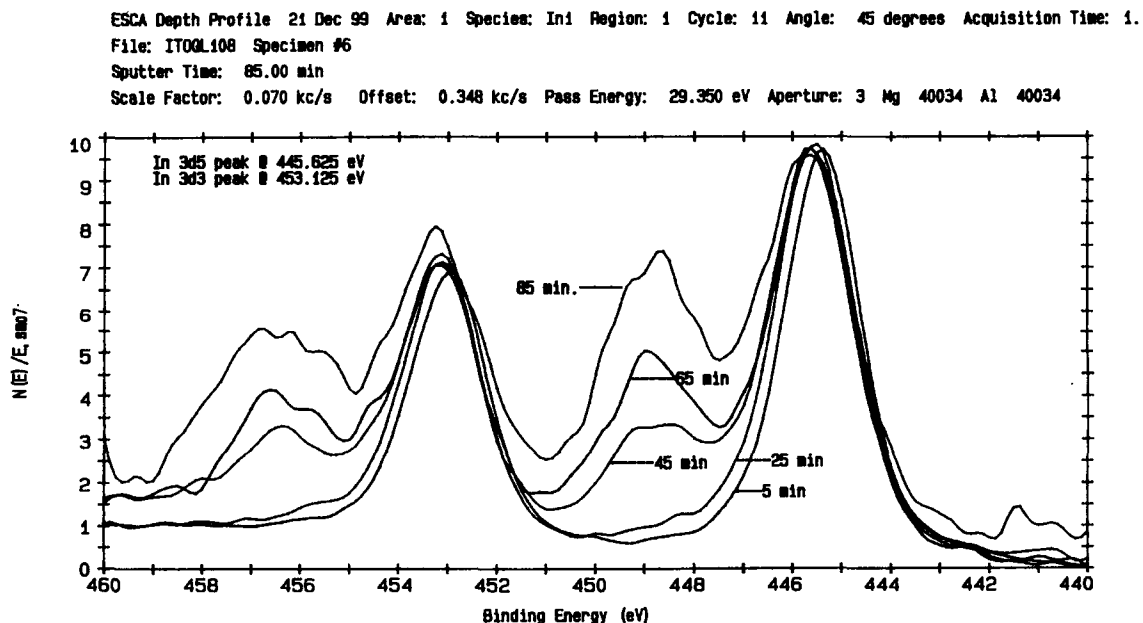


Figure 8. ESCA depth profile of an ITO film deposited onto an alumina film. Note the appearance of the smaller In 3d₅ and In 3d₃ peaks that were 3 eV higher in energy than the In 3d₅ and In 3d₃ peaks (445.6 and 453 eV) in the as-deposited ITO, as the ITO-Al₂O₃ interface was approached. Profile was taken after the films were heat treated at 1400 °C for 2.5 hours.

in the overall stability of the ceramic. As shown in Figure 9, high temperature strain experiments supported these earlier findings in that sensors prepared with aluminum doped ITO active strain elements were responsive at temperatures in excess of 1495 °C. A gage factor of 2.05 was measured for these doped ITO films employed as the active strain elements.

ESCA studies suggest that when aluminum is deposited onto ITO and heat treated to allow diffusion of the aluminum into the surface layers of the ITO, further stabilization of the ITO films is possible. To confirm that aluminum was solely responsible for the enhanced high temperature stability of the sensors, additional depth profiles of ITO films deposited onto aluminum nitride (AlN) films were analyzed by ESCA after heat treatment at 1400 °C for 2.5 hours in air. The additional studies were done to eliminate matrix effects in which the aluminum is bound to help establish the role of aluminum in enhancing the high temperature stability of the ceramic strain sensors; i.e. is there a difference in the stabilizing effect of aluminum when it is bound as an oxide vs. a nitride. An ESCA depth profile of an ITO-AlN interface is shown in Figure 10. Here, the presence of new In 3d₅ and In 3d₃ peaks were observed that were 3 eV higher in energy than the In 3d₅ and In 3d₃ peaks in the as-deposited ITO. This was similar to the ESCA depth profile of the ITO film as the ITO-Al₂O₃ interface was approached. These experiments confirmed that aluminum tends to stabilize the ITO films, regardless of the matrix in which the aluminum was bound. The ESCA studies also confirmed that when aluminum is deposited onto ITO and heat treated to allow diffusion of the aluminum into the surface layers of the ITO, further stabilization of the ITO films is possible as evidenced by the higher bond energies of the indium.

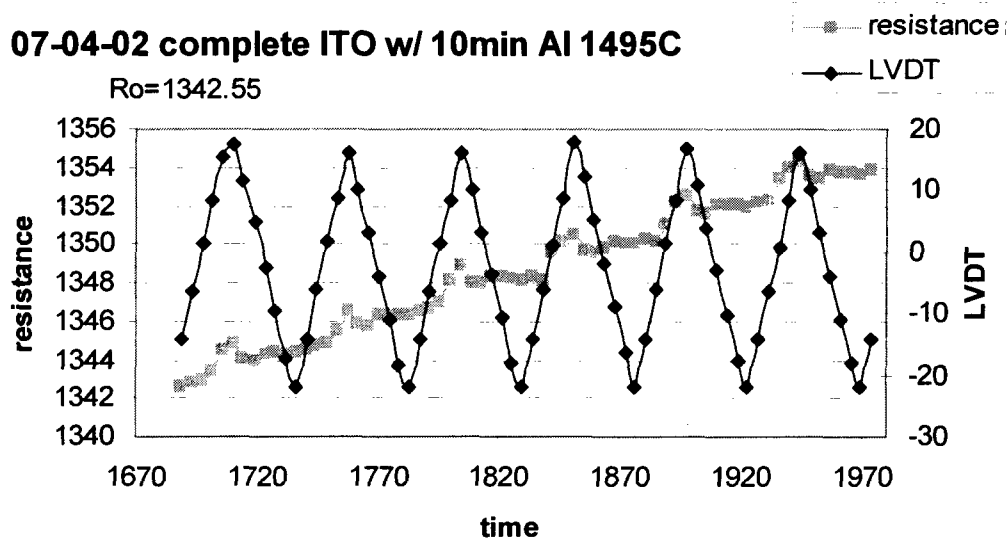


Figure 9. Piezoresistive response of a ceramic thin film strain gage at 1495 °C. The active strain element consisted of an 8 μm thick ITO film onto which a thin aluminum film was deposited to enhance high temperature stability of the sensor. The sensor pattern consisted of serpentine arrays with 100 μm lines and spaces. DR = 0.1073

ESCA Profile 21 Aug 02 Area: 1 Region: 1(In1) Cycle: 120 0 degrees Acq Time: 0.67 m1
 File: ITO105 ITO/AlN 8/2002
 Sputter Time: 59.00 min
 Scale: 0.173 kd/s Offset: 0.504 kd/s Pass E: 58.700 eV Aperture: 3 Al 400 W

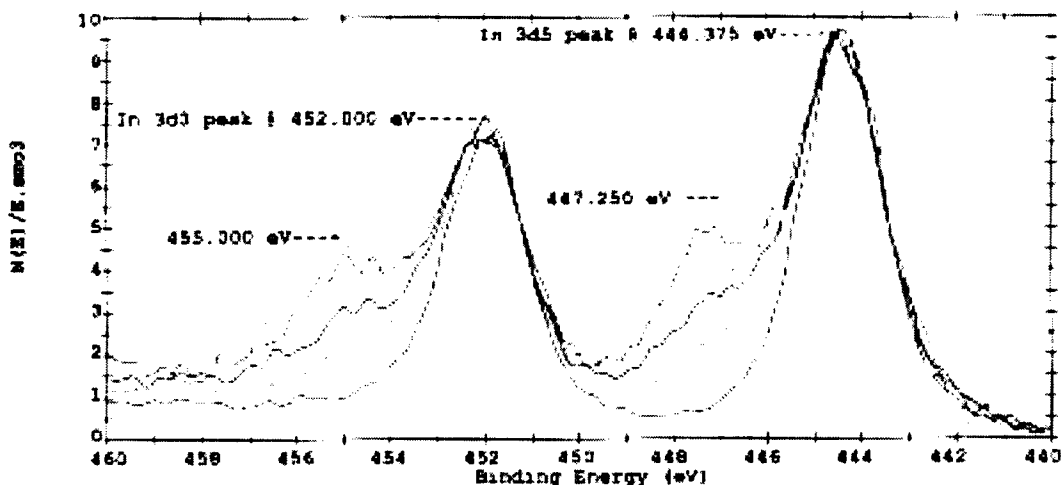


Figure 10. ESCA depth profile of an ITO film deposited onto aluminum nitride film. Note the appearance of the smaller In 3d₅ and In 3d₃ peaks that were 3 eV higher in energy than the In 3d₅ and In 3d₃ peaks (444.375 and 452 eV) in the as-deposited ITO, as the ITO-AlN interface was approached. The ESCA profile was taken after the films were heat treated at 1400 °C for 2.5 hours.

Since the thin film strain gages will ultimately be used to evaluate the mechanical behavior of advanced CMC components, including SiC-SiC and C-SiC CMC's, it is very important to establish an upper use temperature for these sensors. We have established the upper use temperature for a wide range of film thicknesses comprising the active strain element in these sensors and have demonstrated that the high temperature stability of the ITO films comprising the active strain elements in the ceramic strain gages could be substantially improved by doping the ITO with aluminum. A gage factor of 2.05 was measured at temperatures in excess of 1495 °C for the doped ITO films that were prepared by depositing a thin film of aluminum onto the patterned surface of the ITO strain elements and heat treating at 800 °C in low oxygen partial pressure ambient to aid the diffusion of aluminum into the ITO film. Another approach to improve the high temperature stability of the ITO films comprising the active strain elements in the ceramic strain gages was to employ extremely thick ITO films as the active strain elements. By depositing ITO films on the order of 10 µm or more (as opposed to films in the 2–3 µm range), both the high temperature stability and gage factor were improved. The improved stability was evidenced by the lower drift rates at elevated temperature. The results described above, confirm the importance of ITO film thickness and ITO chemistry on the electrical stability and piezoresistive response of ceramic strain gages at temperatures above 1400 °C.

Delivery of instrumented CMC constant strain beams to NASA

Instrumented CMC constant strain beams were delivered to NASA for testing under dynamic strain conditions (high cycle loading). The instrumented CMC beams were fabricated by a combination of shadow masking and photolithography, specifically a liftoff technique that employed a relatively thick polyimide photoresist. The platinum leads and bond pads were fabricated onto a SiC cement layer, stabilized by a thermal sprayed alumina layer. Patterning of the leads was accomplished by sputtering platinum through a shadow mask onto a relatively smooth thermal sprayed alumina layer. The ITO active strain elements were patterned using a lift-off technique, whereby a relatively thick polyimide layer (photoresist layer) was spin cast onto a layer of sputtered aluminum oxide, exposed, baked and developed. This sputtered aluminum oxide layer was deposited directly onto the cement surface to provide stability at elevated temperature (see complete description of bond coat development below). The ITO films were sputtered through windows created in the resist and the excess films were lifted off to delineate the desired patterns. The polyimide photoresist (LOR 50B) manufactured by MicroChem, was spin cast onto the sputtered aluminum oxide layer at a nominal thickness of 6 μm , when spun at 2,200 rpm. The high viscosity LOR 50B photoresist is ideal for patterning the thin film strain gages on the rough surfaces of the SiC-SiC CMC by liftoff techniques, where relatively thick resist layers are required. The platinum lead wires were joined to the platinum films using parallel gap welding. Photographs of the instrumented CMC's complete with wire bonded lead wires are shown in Figures 11 and 12. The active strain elements consisted entirely of ITO and no platinum self compensation circuitry was employed in these sensors, since they were going to be tested under dynamic strain conditions at NASA Glenn's high temperature-high cycle test facility.

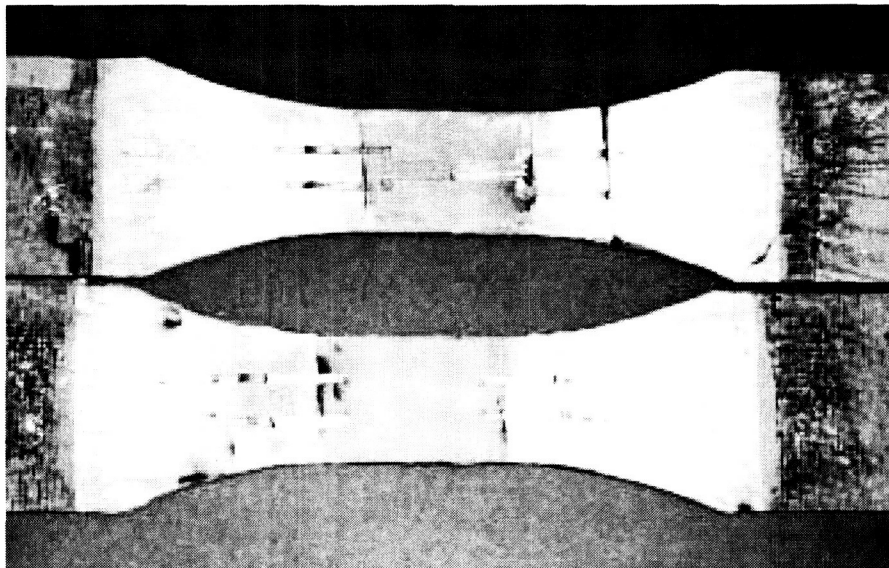


Figure 11. Photograph of instrumented CMC constant strain beams delivered to NASA for testing under dynamic strain conditions (high cycle loading). Note the color of the ITO active strain elements after heat treatment in nitrogen at 700 °C.

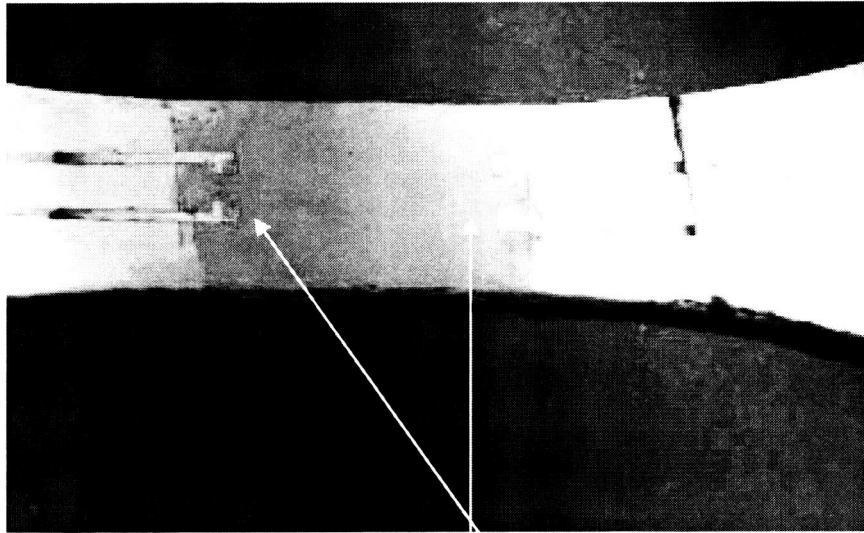


Figure 12. High magnification view of one of the instrumented CMC constant strain beams delivered to NASA for testing under dynamic strain conditions at elevated temperature. Note the grey color of the substrate in the vicinity of the ITO active strain elements as compared to the color of the thermal sprayed alumina layer on either side of the ITO active strain elements. Also, note the orange color of the ITO active strain elements prior to heat treatment.

Development of Bond Coats for SiC-SiC CMC's

A series of bond coats for the SiC-SiC CMC's were developed and tested. The most successful bond coat system developed thus far, allowed conventional lithography and liftoff techniques to be used for strain gage pattern transfer. The bond coats consisted of a proprietary SiC cement supplied by HPI Inc, Ayer, MA onto which a sputtered and/or thermal sprayed alumina layer was deposited. The SiC cement was applied to the oxidized surfaces of the CMC and a sputtered alumina coating (2–5 μm thick) or a thermal sprayed alumina layer (75 μm thick) was deposited onto the SiC cement to further stabilize the cement at elevated temperatures. The purpose of the added ceramic material was to fill in the voids and defects within the surface layers of the CMC so that a conformal surface capable of accepting thin films could be achieved without compromising strength. To prepare the CMC surfaces for these additional ceramic layers, it was necessary to first oxidize the surface for 200 hrs at 1200 $^{\circ}\text{C}$ to form a continuous, protective scale of SiO_2 . The native oxide layer acts to electrically isolate the semiconducting CMC from the active sensor elements. If an alumina layer was not applied to the SiC cement surface, a silicate glass would form on the CMC surface when heated to temperatures in excess of 800 $^{\circ}\text{C}$ as shown in Figure 13. SEM micrographs of this glassy SiC cement layer formed on the CMC coupons after thermal cycling to 1050 $^{\circ}\text{C}$ are shown in Figures 17 and 18. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 $^{\circ}\text{C}$ for 1 hr. After curing, the cement was lapped and another layer of cement was applied and cured. This process was repeated until the surface

roughness was adequate for thin film sensors as shown in Figure 15. An SEM micrograph of the same SiC cement layer after lapping and curing is shown in Figure 19. In some instances, thermal sprayed magnesium aluminate spinel was applied directly to the SiC cement layer and lapped to form a relatively smooth surface. However, the surface roughness was still too great for conventional lithography and liftoff techniques to be used for pattern transfer. Therefore, a sputtered aluminum oxide layer was deposited directly onto the SiC cement surface to provide stability at elevated temperature. It was determined that a 2 μm thick sputtered aluminum oxide layer was sufficient to retard vitrification of the SiC cement when thermally cycled to 1200 °C in air. The reason for this stability is that aluminate glasses do not form at temperatures below 1800 °C. The surface finish of the SiC cement protected by a sputtered aluminum oxide layer was an order of magnitude smoother than the thermal sprayed layer and thus, was much better suited for conventional lithography than the thermal sprayed surface. A photograph of the surface of a sputtered alumina coating on a lapped and cured SiC cement layer is shown in Figure 14.

Recent experiments suggest that a combination of cement layers (a SiC cement layer and an Al_2O_3 cement layer) provides the best surface for sensor attachment in that the surface will be capable of surviving higher temperatures. The reason for this is that aluminate glasses are not readily formed at temperatures below 1800 °C and thus, the SiC cement layer will be stable at temperatures in excess of 1500 °C, the current temperature limitation for the ceramic strain gages. The reaction between the oxidized SiC cement layer and the high purity alumina produces an aluminate that is resistant to vitrification at the temperatures of interest.

Figure 16 shows a bond coat consisting of a combination of SiC cement and alumina cement that was thermally cycled ten times from room temperature to 1200 °C. There was no visible sign of vitrification of the SiC cement. The cement layers are necessary to planarize the rough surfaces of the CMC for attachment of thin film sensors. The CMC surface roughness is not only derived from the SiC weave pattern but is also derived from the considerable porosity in the SiC matrix. Outgassing during the manufacturing of the ceramic matrix is the most likely cause of this porosity. The lapped and cured SiC cement by itself was not a viable option for the deposition of thin film strain gages because of the possibility of forming an electrical short to ground due to its enhanced conductivity at elevated temperature. Also, the discrete SiC particles (partially sintered particles) within the SiC cement were not dense enough to provide an acceptable surface for thin film masking and deposition. One possible reason for the strong bond between the SiC cement and the alumina is the formation of an aluminate layer that developed during high temperature exposure.

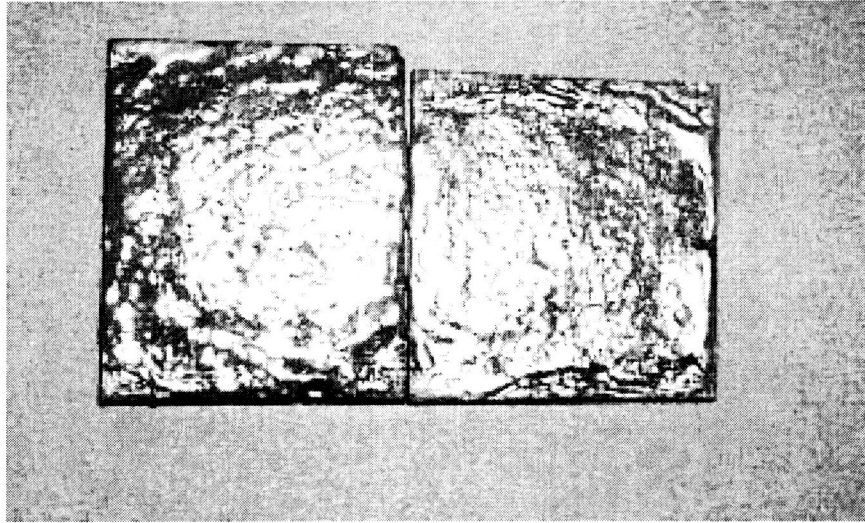


Figure 13. Photograph of SiC cement layer applied to the surface of two SiC-SiC CMC coupons and thermally cycled to 1000 °C in air. Note the glassy material present on the surface of the CMC after exposure. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr.

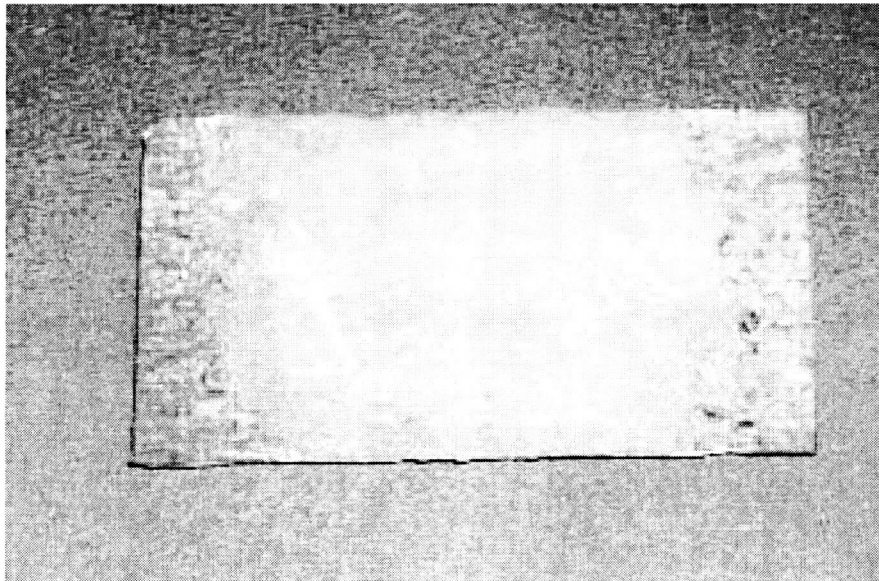


Figure 14. Photograph of SiC cement layer applied to the surface of a SiC-SiC CMC coupon and subsequently coated with a thin film of sputtered alumina. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr. The coupon was thermally cycled from room temperature to 1200 °C in air (10 cycles). There was no evidence of cracking and no glassy layer was evident on the surface of the CMC.

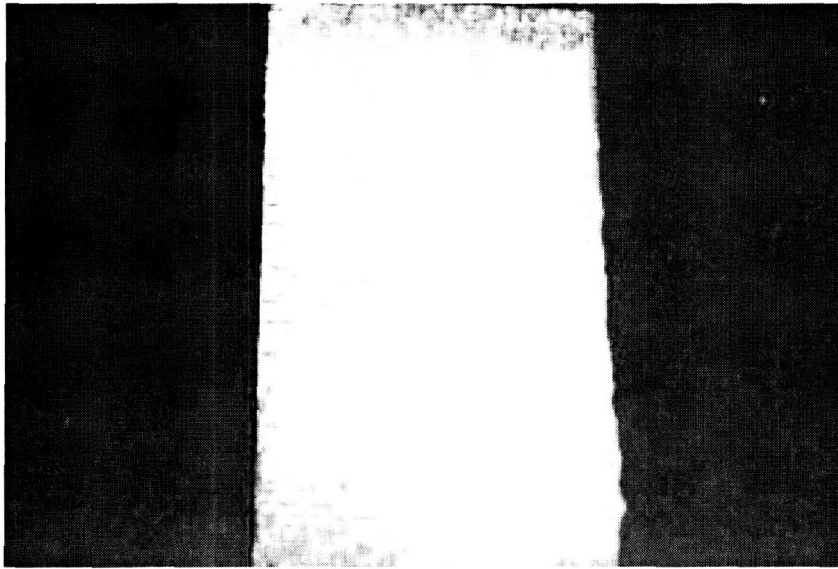


Figure 15. Photograph of a SiC cement layer applied to the surface of a SiC-SiC CMC coupon and subsequently lapped to form an extremely smooth surface. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr.



Figure 16. Photograph of SiC cement layer applied to the surface of a SiC-SiC CMC coupon and subsequently coated with a layer of alumina cement. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr. The coupon was thermally cycled from room temperature to 1200 °C in air (10 cycles). There was no evidence of cracking and no glassy layer was present on the surface of the CMC.

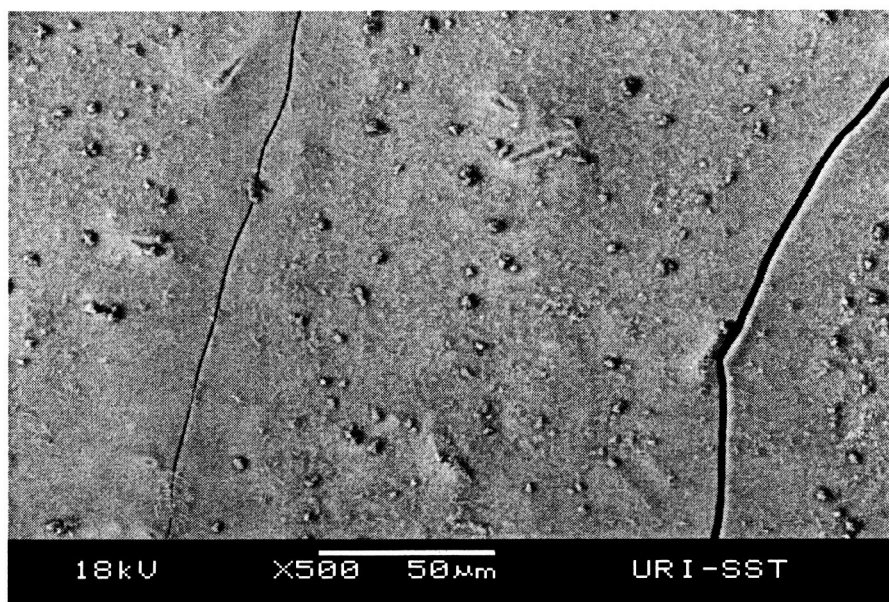


Figure 17. SEM micrograph of a SiC cement layer applied to the surface of a SiC-SiC CMC coupon and thermally cycled to 1000 °C in air. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr. Note the densification of the surface relative to the as-cured material in Figure 19 and the vitrified structure of the glassy layer. Extensive cracking of the glass after thermal cycling is also evident.

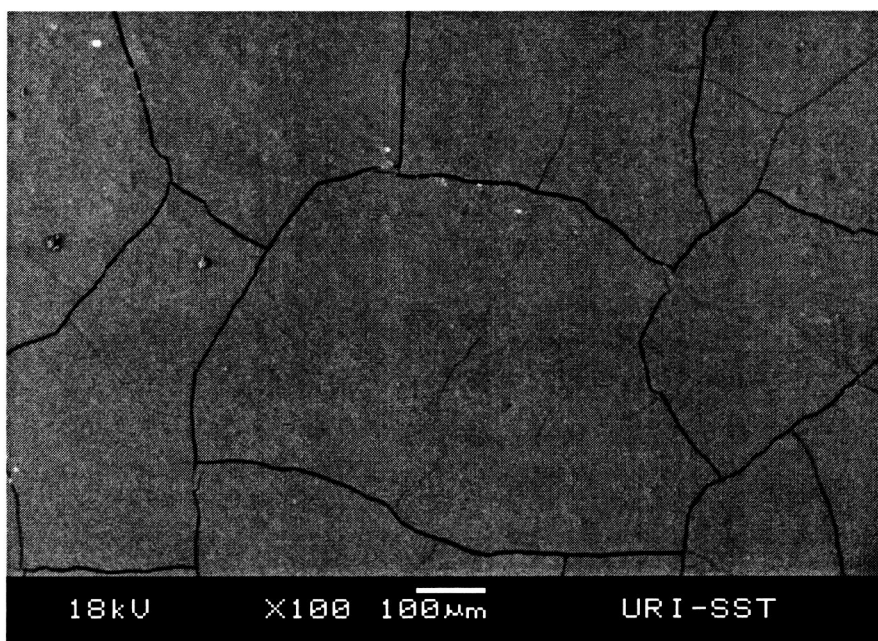


Figure 18. SEM micrograph of the surface of a SiC cement layer applied to a SiC-SiC CMC coupon and thermally cycled to 1000 °C in air. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr. Note the densification of the surface relative to as-cured material and the vitrified structure of the glassy layer. Extensive cracking of the glass after thermal cycling is also evident.

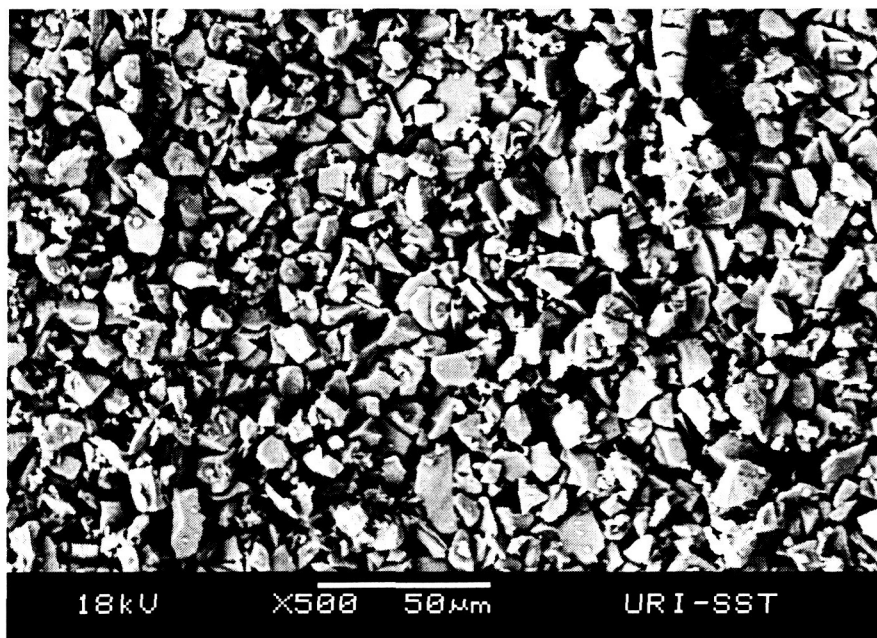


Figure 19. SEM micrograph of the surface layer of SiC cement applied to the surface of a SiC-SiC CMC coupon and cured. The SiC cement was applied by a tape-casting (doctor blade) technique and cured at room temperature for 24 hrs followed by a high temperature cure at 400 °C for 1 hr.

Fabrication of sensors on Alumina and SiC-SiC CMC Constant Strain Beams

Since one of the goals of this program was to fabricate ceramic thin film strain gages on advanced ceramic matrix composites (CMC's), considerable effort was made to develop a lithographic process that would be compatible with the rough surfaces of the CMC's without compromising performance and resolution (line width and line spaces) of the strain gage elements. This surface roughness required extensive surface preparation of the CMC's to insure adhesion of the thin film sensors and yet provide adequate electrical isolation so that shorting to ground at elevated temperatures will not occur. Since these thin film devices will ultimately be used to evaluate the mechanical behavior of advanced SiC-SiC CMC components, a robust sensor capable of operating under severe environmental conditions was required. Towards this end, a number of thermal sprayed coatings were developed. These thermal sprayed coatings consisted of high purity alumina and/or a magnesium aluminate spinel. The coatings were deposited onto specially prepared CMC surfaces in an attempt to fill the voids and defects within the surface layers of the CMC, so that a conformal surface capable of accepting thin films could be achieved without compromising the strength and integrity of the CMC surface layers. All thermal sprayed coatings were supplied by HPI Inc, Ayer, MA. An alternative to thermal spraying is to remove material from the surface layers of the CMC but this would have an adverse affect on the mechanical integrity of the outermost fibers of the CMC material. Prior to the deposition of these thermal sprayed layers, it was necessary to oxidize the surface of the CMC for 200 hrs at 1200 °C to form a continuous,

protective scale of SiO₂ over the individual fibers in the SiC weave. The native oxide formed on the SiC surface was used to isolate the active sensor elements from the electrically conductive CMC, in the event that the added thermal sprayed layers become compromised at very high temperatures ($T \approx 1500$ °C).

Small SiC-SiC CMC process monitors were used to develop a robust lithography technique for the CMC constant strain beams supplied by NASA. A combination of patterning techniques including shadow masking and lift-off lithography were used for this purpose. In locations where dimensions are not critical, such as lead films and bond pads, shadow masking was used for pattern transfer. In locations where dimensions were more critical, such as the active strain elements, lithography (a lift-off technique) was used. Lead wires were attached to the thin film bond pads after completion of the sensors. In a separate study, the merits of laser welding to attach lead wires to the films are being explored instead of parallel gap welding to make more robust joins and eliminate excessive heating in the regions adjacent to the join.

Due to the extreme surface roughness of the CMC substrates, new lithography techniques were developed whereby a uniform layer of polyimide resist (LOR 10B) was applied under viscous flow conditions (gravity flow) while being heated at 60 °C. This allowed enough solvent to evaporate during spreading of the resist to prevent blistering of the resist during subsequent heating cycles and ultimately proved to be very successful. It was determined that resist layers on the order of 10–60 µm thick were sometimes necessary for complete coverage of the thermal sprayed CMC surface. This required up to 4 layers of 15 µm thick polyimide resist followed by two layers of a conventional spin cast positive resist to achieve the necessary coverage of the rough surfaces and permit the patterning of the sensor using liftoff. Multiple layers of the polyimide resist could be uniformly deposited over the surface of the CMC without trapping excess solvent by gravity spreading. Several sensor patterns were lithographically transferred using this newly developed lift-off process. Specifically, the modified lift-off process employed a bilevel polyimide-based photoresist (LOR 10B, MicroChem Inc.) and a conventional positive photoresist (Shipley, Inc). This bilevel polyimide resist permitted both ceramic films such as ITO and metal films such as platinum to be rf sputtered through windows created in the resist without excessive damage to the resist.

All lithographic processes employed a polyimide-based photoresist (MicroChem Inc.) in conjunction with a modified lift-off process to delineate the desired sensor patterns for the self-compensated strain gages shown in Figures 20–26. This polyimide resist is synthesized by high pressure imidization of PMMA in ammonia to form polymethylglutarimide (PMGI). These PMGI resists have much greater thermal stability than conventional photoresists as well as a higher glass transition temperature, making it capable of withstanding prolonged exposure to rf plasmas and their undesirable electron heating effects. Typical bake temperatures for these resists are on the order of 175 °C and are supplied by the manufacturer in a wide range of viscosities so that final resist thickness can be as great as 6 µm (LOR 50B) or as little as 0.5 µm (LOR 7B) at typical spin speeds (2,000 rpm). The LOR 7B, LOR 10B and LOR 50B polyimide resists are typically applied to a surface by spin casting and are baked at relatively high temperatures due to their high

glass transition temperatures, before applying the imaging resist (Shipley SC1827) that delineates the final pattern. A typical application of the polyimide resist consists of spin casting at 1500–2500 rpm, followed by soft baking at 105 °C for 2 min and additional hard baking at 155 to 175 °C for 8 min. After hard baking, an additional positive resist or imaging resist (Shipley SC1827) is spin cast at 2500 rpm onto the polyimide resist and soft baked at 95 °C for 3 min. A photomask containing the desired artwork is placed over the resist-coated substrate and exposed to UV light (5 min exposure) to create the desired pattern in the resist. The image is developed using an AZ developer, which dissolves both the exposed imaging resist and underlying polyimide resist. After development, the ITO and/or platinum films are sputtered through the windows created in the resist-coated substrate. After deposition, the substrate is placed in a PMGI resist stripper (manufactured by MicroChem Inc.) to remove the excess films from the substrate. The entire process is repeated for the deposition of each film (and pattern) type to generate the completed self-compensated strain gage pattern.

Sensor design and fabrication of small footprint sensors on alumina and SiC-SiC CMC's.

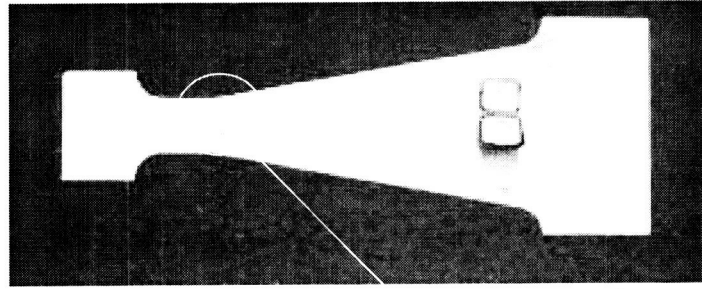
The PMGI polyimide resist permits both ceramic films such as ITO and metal films such as platinum to be rf sputtered through windows created in the resist without excessive damage to the resist, i.e. the resist will not flow due to the excessive heating. This is particularly important when long deposition times (and long exposure times to the rf plasma) are required to keep the resistance of the small footprint sensors low. Thus, the PMGI resist enables small footprint devices (narrow line widths and line spaces) to be fabricated by rf sputtering. Deposition of the sensor materials by rf sputtering provides much better adhesion at elevated temperatures than does dc sputtering and thus, the reason for using this deposition method. The heating effects associated with rf sputtering often precludes the use of commonly used photoresists in these applications. The PMGI resists used by us are compatible with conventional positive photoresists (Shipley SC1827) in that the exposure and development steps will affect both layers equally. However, the PMGI layer must be applied first, soft baked and hard baked in separate steps adding slightly to the complexity of the lithography process. The reason for this is that mixing of the positive (imaging) resist with the polyimide can occur during spin casting, if the polyimide is not baked for sufficient time at temperature; i.e. if the solvent is not removed completely during the baking of the polyimide resist, dissolution of the polyimide in the imaging resist can occur. The lift-off process described above is capable of submicron resolution and is greatly facilitated by the enhanced thickness of the PMGI resist layer and associated undercutting of the PMGI resist layer during development. A micrograph of a self-compensated thin film strain gage deposited on alumina using the bilevel positive resist in conjunction with liftoff is shown in Figure 20. The self-compensated circuitry shown in detail in Figure 20 consisted of a platinum serpentine array with 100 μm lines and spaces and a 100 μm ITO line. A similarly prepared uncompensated ITO strain gage (ITO element is transparent) with 100 μm lines and spaces is shown in Figure 21 (a). This sensor was processed in the same manner as above but the serpentine array consisted entirely of ITO; i.e., no platinum

self-compensation circuitry was employed. The uncompensated ITO sensor shown in Figure 21 (a) was tested at 1450 °C and exhibited excellent piezoresistive response and an extremely low drift rate at this temperature. This sensor remained operational at temperatures in excess of 1480 °C in air, yielding a gage factor of 2.5. The bond pads and thin film leads in this sensor were made of platinum and the active strain element was made of ITO.

An uncompensated ITO strain gage with 100 μm ITO lines and spaces is shown in Figure 21 (b). The active ITO strain element (almost transparent in the photograph) was prepared using a liftoff process with a bilevel positive resist (PMGI polyimide resist in combination with a Shipley imaging resist). A self-compensated ITO sensor shown in Figure 22, was fabricated on an alumina constant strain beam in a similar manner and tested at temperatures in excess of 1480 °C in air. The bond pads and leads to the ITO active strain elements were made of platinum and provided self-compensation attributes to the sensor. This self-compensated thin film strain gage had the smallest footprint of all self-compensated strain gages fabricated to date and consisted of 75 μm platinum lines and spaces. It was fabricated on an alumina constant strain beam using a liftoff process with a bilevel positive resist (PMGI resist in combination with a Shipley imaging resist). Even though this sensor had an extremely small footprint, it exhibited a large piezoresistive response with extremely low drift rate.

To uniformly coat the rough surfaces of the CMC's, multiple layers of the LOR 10B had to be applied under viscous flow conditions (gravity flow) while being heated at 60 °C. In this way, the PMGI polyimide resist could conformally coat the entire CMC without trapping excess solvent. Prior to application of the LOR 10B, the surfaces of the CMC were coated with a layer of SiC cement and stabilized with a thermal sprayed alumina layer (75 μm thick). After multiple applications of LOR 10B, a conventional positive photoresist (Shipley, Inc) was applied to the surface by spin casting. The final sensor patterns were lithographically transferred using a lift-off process, as shown in Figures 23 (a) and (b). The combination of features from the SiC cement and the thermal sprayed alumina provided a bonding layer that could be lapped to a very smooth surface. After removal of the excess films, the thermal sprayed surfaces were cleaned in an oxygen plasma to remove all residual organic films. With the PMGI polyimide resist, both ceramic films such as ITO and metal films such as platinum could be rf sputtered through windows created in the resist without excessive damage to the resist. However, the small footprint sensors were difficult to transfer onto this surface.

To produce sensor patterns on CMC's with finer geometries than those possible with a SiC cement layer used in combination with a thermal sprayed alumina layer as shown in Figures 23 (a) and (b), a better finish was required. To accomplish this, SiC cement was applied to the oxidized surfaces of the CMC and a sputtered alumina coating (2–5 μm thick) was deposited onto the SiC cement to further stabilize the cement at elevated temperatures. The surface roughness was improved by more than an order of magnitude using this method and enabled single layers of LOR 50B polyimide resist to be spin cast. The LOR 50b was applied to the specially prepared CMC surfaces by spin casting and



Strain sensor 2

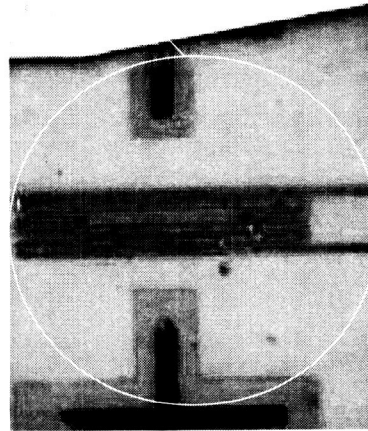


Figure 20. Optical micrograph of a self-compensated thin film strain gage deposited on alumina using a bilevel (polyimide) positive resist in conjunction with liftoff. The sensor pattern consisted of serpentine arrays with 100 μm lines and spaces. Note the yellow color of the ITO strain element in the center of the serpentine pattern made of platinum.

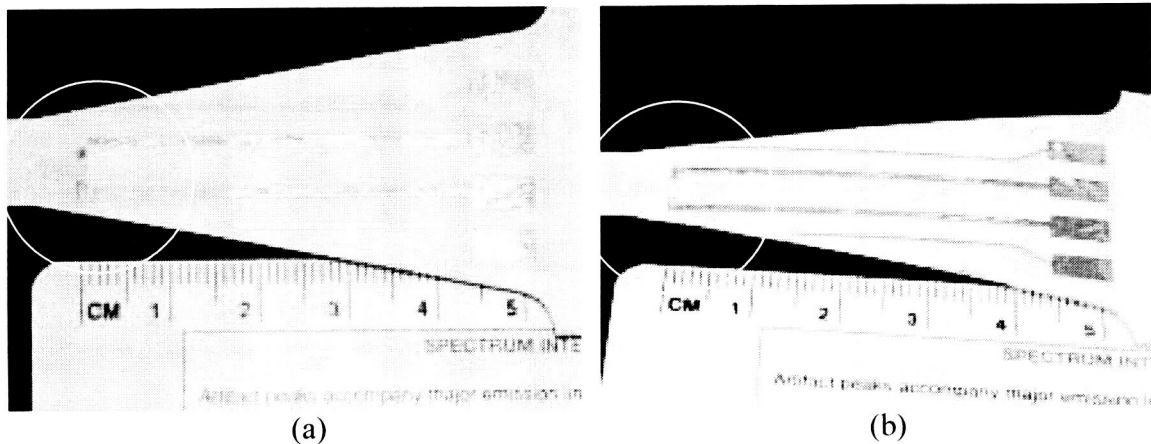


Figure 21. (a) Photograph of an uncompensated ITO strain gage (ITO element is transparent) with 100 μm lines and spaces. This alumina constant strain beam and sensor were tested at temperatures in excess of 1480 $^{\circ}\text{C}$ in air. The bond pads and leads are made of platinum. Gage factors of 2.5 were realized at temperatures of 1450 $^{\circ}\text{C}$, with higher gage factors observed in the temperature range 1200 to 1400 $^{\circ}\text{C}$. (b) Photograph of an uncompensated ITO strain gage (ITO element is transparent) with 100 μm lines and spaces. This alumina constant strain beam and ITO sensor were tested at temperatures in excess of 1450 $^{\circ}\text{C}$ in air. The bond pads and leads are made of platinum.

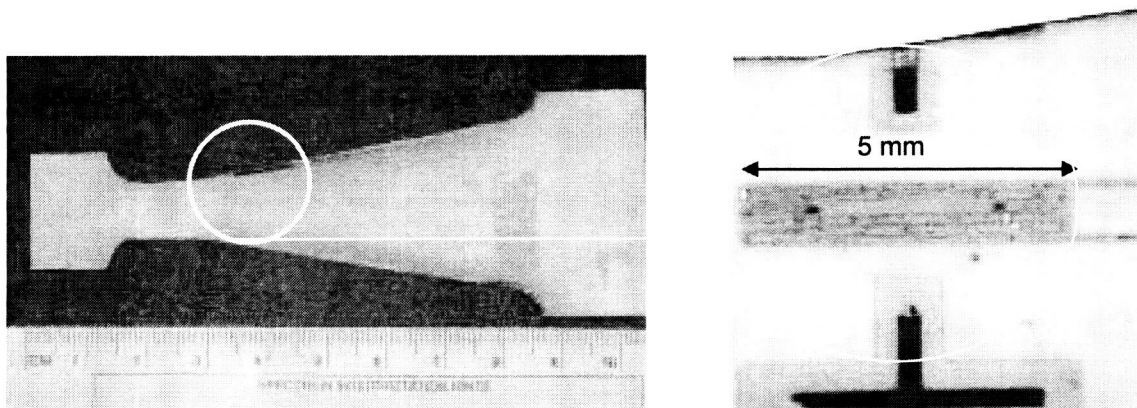


Figure 22. Optical micrograph of a self-compensated thin film strain gage deposited on an alumina constant strain beam. The sensor was fabricated using a liftoff technique in conjunction with a bilevel positive resist (PMGI polyimide resist). The self compensation circuitry consisted of 75 μm platinum lines and spaces. The active ITO strain element in this self compensated strain gage is located in the center of the serpentine array (narrow yellow line).

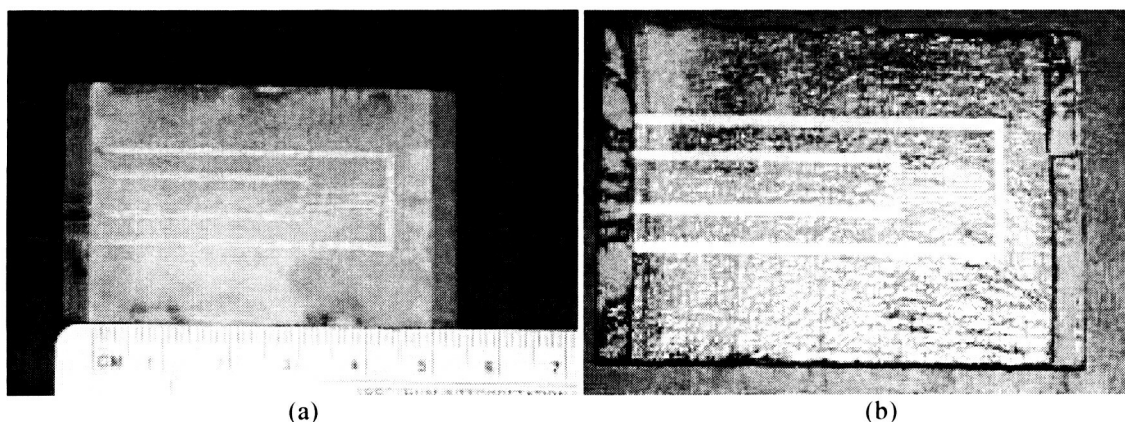


Figure 23. (a). Photograph of a completed thin film strain gage on a CMC substrate that was prepared using a combination of SiC cement and thermal sprayed alumina layers. The sensor was thermally cycled ten times from room temperature to 1200 $^{\circ}\text{C}$. (b) Photograph of the self-compensated thin film strain gage pattern (a) formed on a SiC-SiC CMC using a bilevel (polyimide) positive resist. Note the color of the thermal sprayed alumina exposed underneath the LOR 10B resist. The self-compensation circuitry consisted of 200 μm platinum lines and spaces.

the patterns were lithographically transferred using a lift-off process, as shown in Figures 24 (a) and (b). A typical process used to pattern the alumina and CMC constant strain beams using lithography in conjunction with lift-off is as follows: (1) rinse surface with THF (tetrahydrofuran), blow dry w/ nitrogen, bake @ 150 °C for 5 min and spin-coat LOR 10B (or LOR 50B) @ 2000 rpm for 1 min; (2) bake @ 155 °C for 1 min; (3) spin-coat Shipley 1827 @ 2000 rpm for 1 min and bake @ 100 °C for 1 min; (4) expose to UV for 7.5 min; (5) develop in AZ Developer and bake @ 100 °C for 1 min; (6) ozone clean (oxygen plasma) @ 100 watts for 15 min; (7) sputter either ITO and/or platinum films; (8) lift-off excess films using MicroChem nanoremove.

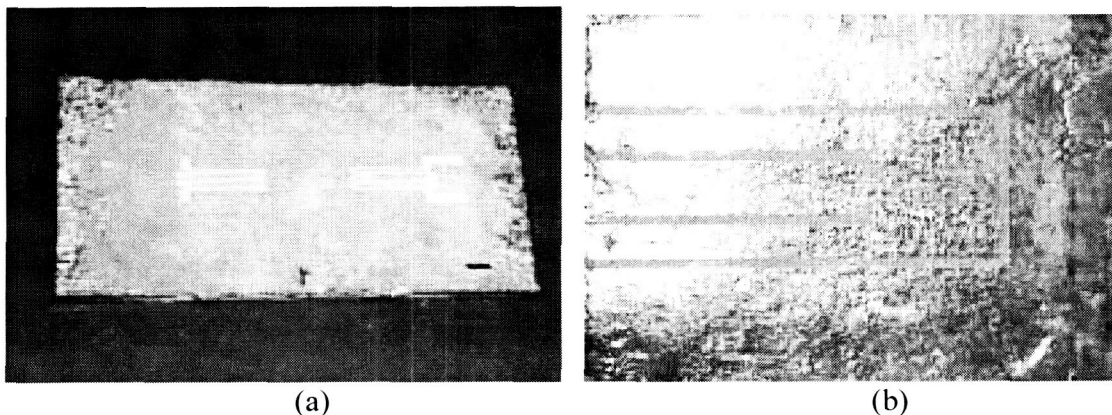


Figure 24. (a). Photograph of two thin film strain gage patterns on a CMC substrate prepared with a combination of SiC cement and sputtered alumina. (b) Photograph of a self-compensated thin film strain gage pattern formed on a CMC substrate prepared with a combination of SiC cement and sputtered alumina. Note the SiC cement/sputtered alumina lines visible underneath the LOR50B photoresist that covers the bulk of the CMC coupon. The self-compensation circuitry consisted of 200 μm platinum lines and spaces.

The best surface finish for the attachment of thin film sensors to date was achieved by a combination of two cement layers; a SiC cement layer applied by a doctor blade technique and an Al_2O_3 cement layer applied by airbrushing. This combination of cements resulted in a surface finish that was an order of magnitude smoother than that achieved with a lapped thermal sprayed layer of alumina but was much better suited for conventional lithography than the sputtered alumina surface. The reason for this is that the aluminum phosphate binder in the Al_2O_3 cement can fill the porosity in the SiC cement layer and thus, seal the surface to permit conventional lithographic processes to be used. The LOR 50B photoresist is not readily drawn into the pore structure of the underlying SiC cement layer by capillarity and thus, permits the efficient transfer of the strain gage patterns without trapping excess solvent. Recent experiments suggest that this combination of cement layers (SiC cement and Al_2O_3 cement) provides an excellent surface for sensor attachment in that the surface will be capable of surviving higher temperatures than those possible with the sputtered alumina films. A CMC coupon

prepared with a SiC cement layer applied by a doctor blade technique and an Al_2O_3 cement layer applied by airbrushing after which an uncompensated ITO sensor pattern was transferred to the CMC using conventional lithographic processes is shown in Figure 25 (a). The completed ITO strain gage transferred to the CMC surface after liftoff is shown in Figure 25 (b).

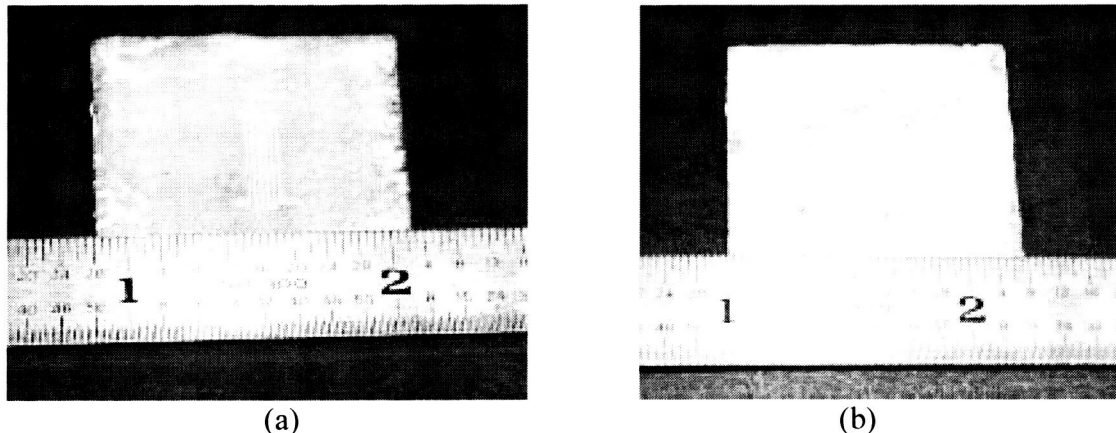


Figure 25. (a) photograph of an uncompensated ITO strain gage pattern transferred to a CMC coupon whose surface was prepared with a SiC cement layer and an air brushed Al_2O_3 cement layer. (b) photograph of a completed ITO strain gage transferred to a CMC surface after liftoff.

A number of new sensor designs were generated using CAD and were transferred to mylar by Southwest Image Inc., Phoenix, AZ, to produce appropriate photomasks for lithography. These included a number of self-compensated and uncompensated ITO strain gage patterns, the geometries of which varied from 400 μm lines and spaces down to 50 μm lines and spaces. In addition, several NASA PdCr strain gage patterns with triangular serpentine arrays were redesigned by us so that the bond pads and thin film leads could fit within the reduced cross sectional area (constant strain area) of the CMC beam and yet have geometries large enough to be compatible with the surface modifications necessary for transferring the strain gage patterns to the rough CMC surfaces. The original NASA PdCr strain gage patterns with triangular serpentine arrays consisted of geometries (20 μm lines and spaces) that were too small to be transferred to the CMC surfaces by conventional lithography. The revised CAD designs of the NASA PdCr strain gage were transferred to mylar by Southwest Image, Inc. and have been processed into completed sensors. A NASA PdCr strain gage pattern transferred to a CMC coupon whose surface was prepared with a SiC cement layer and an air brushed Al_2O_3 cement layer is shown in Figure 26.

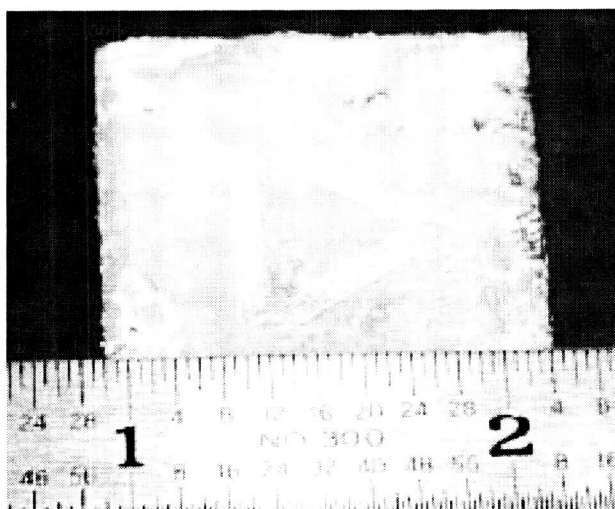


Figure 26. Photograph of a NASA PdCr strain gage pattern transferred to a CMC coupon. The surface was prepared with a SiC cement layer and an air brushed Al_2O_3 cement layer.

Due to the surface roughness of the CMC's, very thick photoresist layers were required to minimize the formation of pinholes and other defects in the resist coatings so that the unwanted sputtered material would not be deposited onto the CMC as shown in Figure 23(a). Several techniques including gravity spreading under viscous flow conditions and silk screening were used to achieve thick polyimide coatings but these proved to be unsuccessful due to the difficulty in removing excess solvent during the soft bakes. Multiple spin castings of the high viscosity PMGI polyimide resists (LOR 50B) at high spin rates proved to be the most successful method in achieving the desired thick polyimide coating thickness. To further improve the lithography and liftoff procedures described above, micro-contact printing using PDMS stamps was investigated to transfer the thin film strain gage patterns to the resist coated substrates. This approach has great potential for transferring the thin film strain gage patterns to extremely rough surfaces, without losing resolution. Micro-contact printing employing PDMS stamps and pattern transfer using soft lithography showed considerable promise in their ease in producing a large number of sensors on rough, curved surfaces in a relatively short period of time.

The materials used to make the PDMS stamps required in the soft lithography process included; polydimethylsiloxane (PDMS)-Sylgard silicon elastomer 184 and Sylgard curing agent 184 (Dow Corning Corp. Midland, MI), polyimide resist material (LOR50B) and Shipley 1827 positive photoresist. The procedure to make the stamps is as follows: (1) conventional photolithography was used to fabricate the sensor pattern on an alumina substrate or master, (2) a 10:1 (by volume) mixture of PDMS and its curing agent is poured onto the patterned alumina (master) and the PDMS is cured at room temperature for 30–60 min. followed by a 1 hour bake at 50C, (3) the PDMS is cast against the polyimide (PMGI) as a replica mold to duplicate the pattern present on the surface of the alumina substrate, (4) the PDMS is carefully peeled off the PMGI mold, (5) the substrate was washed in methanol and acetone, followed by N_2 blow dry, (6) the substrate was baked at 170 °C, 30 mins, (7) spin-cast LOR50B 3500 rpm on substrate and bake for 3 mins, (8) sputter 10–20 nm of copper on the LOR50B coated substrate, (9) a cotton applicator was dipped into a 0.01M thiol solution (diluted by alcohol) and brushed onto

the PDMS stamp, (10) PDMS stamp was brought into contact with the substrate for 10 seconds, (11) the substrate was then etched in 0.5M FeCl₃ solution and cleaned with DI water, (12) the substrate was placed in AZ developer to remove the PMGI resist in the copper windows created by etching in FeCl₃ and (13) the thin films are sputtered through the windows created in the PMGI resist and lifted off using MicroChem nanoremove.

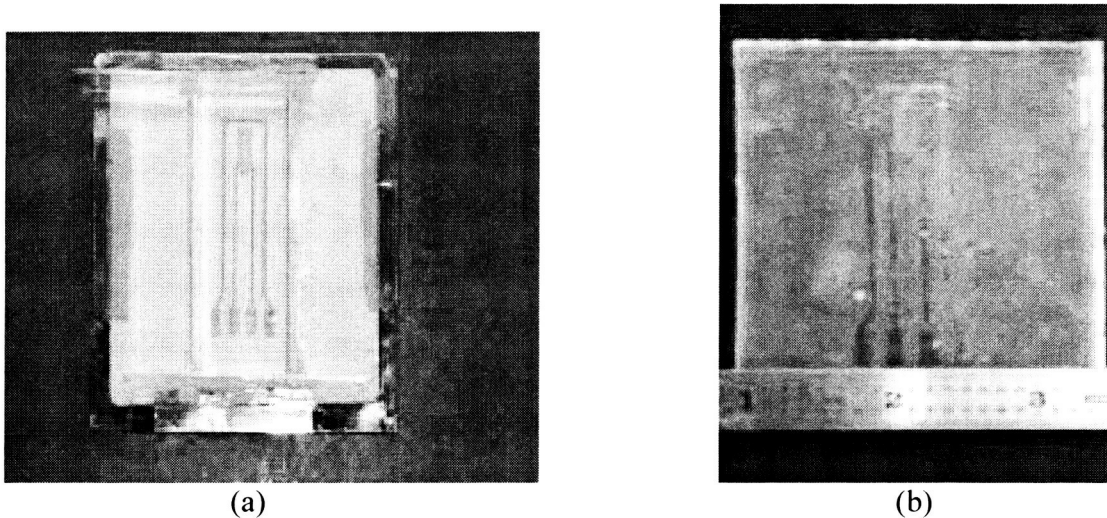


Figure 27. (a) photograph of the mold used to form a PDMS stamp with a thin film strain gage pattern. The pattern shown on the left was fabricated on an alumina master and transferred by photolithography. (b) photograph of a PDMS stamp with the thin film strain gage pattern shown on the left.

Publications: NASA APP “Ceramic strain gages for use at temperatures up to 1500 °C”:

1. Materials Research Society Symposium Proceedings (Symposium Z), Boston, MA, “Stabilization of Indium Tin Oxide Films at Very High Temperatures”; O.J. Gregory, T. You, E.E. Crisman and M. Platek, December 2002.
2. USAF Range Commanders Council-Twentieth Transducer Workshop Dayton, OH “Ceramic Strain Gages for Use at Temperatures Approaching 1450 °C,” O.J. Gregory, Q. Luo and E.E. Crisman, June 18–19, 2002.
3. O.J. Gregory, Q. Luo, J. Bienkiewicz, B.M. Erwin and E.E. Crisman, “An Apparent “n” to “p” Transition in Sputtered ITO High Temperature Strain Gages,” Thin Solid Films, 405, p. 263–269, (2002).
4. O.J. Gregory, Q. Luo, and E.E. Crisman, “High Temperature Stability of Indium Tin Oxide Thin Films,” Thin Solid Films, 406, p. 286–293, (2002).

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Conclusion

Indium-tin-oxide (ITO) thin film strain gages were successfully demonstrated at temperatures up to 1500 °C. These laboratory tests in air indicated that relatively thick ITO strain gages produced a stable piezoresistive response with little drift over the temperature range 25 °C to 1500 °C. These high temperature static strain tests revealed that the piezoresistive response and electrical stability of the ceramic sensor depend largely on the thickness of the ITO films comprising the active strain elements. When relatively thin ITO films (2.5 µm thick) were employed as the active strain elements, the piezoresistive response was reasonably large at lower temperatures (1157 °C) but became unstable at temperatures above 1225 °C. In contrast to this, ceramic sensors prepared with 5 µm of ITO showed a gage factor 4.94 and an upper temperature limit of 1430 °C, whereas 8 µm of ITO survived 20 hours of exposure at 1481 °C with a gage factor 2.36 and drift rate of 0.0092/hour. Very thick ITO strain gages (10 µm) permitted the sensors to operate beyond 1500 °C. Based on these results, we have concluded that the thickness of the ITO active strain element plays a significant role in the high temperature stability of these ceramic strain gages. Thicker ITO films appear to be effective oxygen diffusion barriers, slowing the migration of oxygen to the platinum: ITO interface and thus, stabilizing the ohmic contact formed between the platinum and ITO. To further improve the high temperature stability of the ceramic strain gages, the active ITO strain elements were doped with aluminum and heat treated to allow interdiffusion of the aluminum into the surface layers of the ITO. ESCA depth profile showed that there is an interfacial reaction between the ITO films and alumina resulting in higher bonding energies associated with the indium-indium bonds.

Instrumented CMC constant strain beams were delivered to NASA. Platinum leads and ITO thin film strain gages were fabricated onto oxidized CMC surfaces onto which a SiC cement layer was applied and further stabilized by a thermal sprayed alumina layer. Patterning of the leads and bond pads was accomplished by sputtering platinum through a shadow mask onto a relatively smooth thermal sprayed alumina layer. The ITO active strain elements were patterned using conventional lithography and a lift-off technique. Due to the extreme surface roughness of the CMC substrates, new lithography techniques and surface preparation methods were developed. These developments were critical to the patterning of small footprint thin film sensors. The best surface finish to date for the attachment of thin film sensors was achieved by a combination of cement layers; a SiC cement layer applied by a doctor blade technique and an Al₂O₃ cement outer layer applied by airbrushing. This combination of cements resulted in a surface finish that was an order of magnitude smoother than that achieved with a lapped thermal sprayed alumina and was much better suited for conventional lithography. Micro-contact printing using PDMS stamps was also investigated to transfer the thin film strain gage patterns to the resist coated CMC substrates. This approach has great potential for transferring the thin film strain gage patterns to the extremely rough surfaces associated with the CMC's.

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